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OPTICALLY INDUCED TRANSFORMATIONS OF METAL TCNO  
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E I KAMITSOS ET AL. 17 SEP 82 TR-82-02 N00014-75-C-0883

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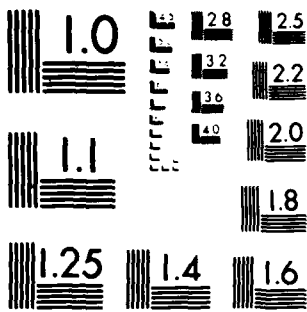


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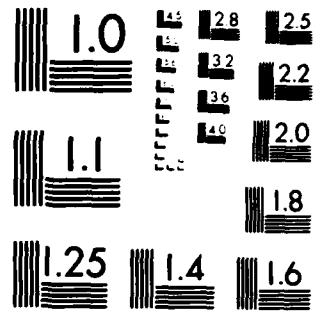
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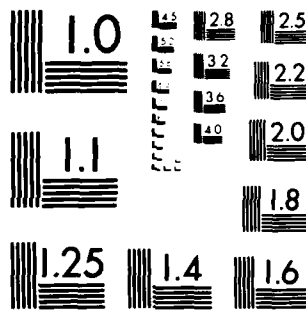
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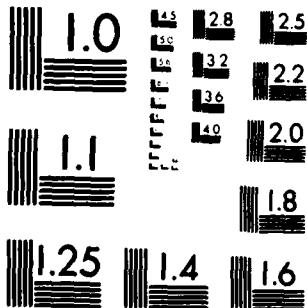
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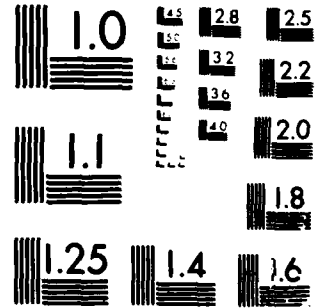
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Optically Induced Transformations of Metal TCNQ Materials

by

E. I. Kamitsos and W. M. Risen, Jr.

Prepared for Publication

in

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Photoinduced transformations of CuTCNQ and AgTCNQ films, and several metal TCNQ salts with visible light have been observed and studied by Raman spectroscopy. Both partial and complete transformations, leading to metal and TCNQ in various material forms, are effected, and implications for photo-switching and light writing discussed.		

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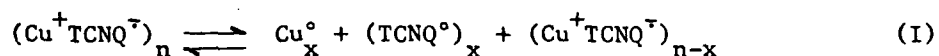
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# Optically Induced Transformations of Metal TCNQ Materials

Efstratios I. Kamitsos and William M. Risen, Jr.

## 1. INTRODUCTION

Recently we showed (1) that the mechanism of the reported electric field-induced transformation of CuTCNQ films from a high impedance to a low impedance state (2) can be elucidated by vibrational spectroscopy. Raman data showed that immediately after the material is electrically switched to the low impedance form about 10-15 mole percent of the TCNQ moieties were in the TCNQ<sup>o</sup> form, and that nearly 40 mole percent were in the TCNQ<sup>o</sup> form after an  $\sim 1.7 \times 10^6 \text{ Vm}^{-1}$  field was applied for 25 hr. This is consistent with the proposed reaction (1,2):



Among the factors that were central to obtaining this result were careful control of the power of the incident Raman source to less than threshold values, preparation of high quality films by vapor deposition and thermal techniques, and acquisition of valid infrared data. The CuTCNQ is the thermally stable form, and (I) does not proceed with heating.

The control of the power of the laser Raman source is essential, because, as we report here, CuTCNQ and several of its analogs, AgTCNQ, Cu(TCNQ)<sub>2</sub> and LiTCNQ, exhibit light-induced transformations (3). These transformations occur when the metal TCNQ materials are irradiated with visible light above threshold powers, whose values depend on the compound, its specific form, and the wavelength of light. The materials studied include uniform homogeneous films, prepared by vapor deposition and thermal treatment, polycrystalline films formed by dipping the metal into a TCNQ solution, and powders prepared by solution reactions.



The degree to which the transformations proceed depends on the power of the light, the duration of exposure, and other factors. Key results are communicated below for two cases; partial transformations, which are reversible in the CuTCNQ, AgTCNQ and  $\text{Cu}(\text{TCNQ})_2$  cases, and complete transformations, which result when the irradiation is carried out under conditions such that TCNQ can escape and the metal remain. The partial transformation of CuTCNQ and AgTCNQ yields materials which are spectroscopically identical to the low impedance form of each, and can lead to photoswitching from the high to low impedance form. The complete transformations lead to light writing and formation of metallic regions in the exposed areas, making the original films function as optical beam resists.

## 2. EXPERIMENTAL

The CuTCNQ, AgTCNQ,  $\text{Cu}(\text{TCNQ})_2$  and LiTCNQ powders were prepared by the method of Melby, et al (4). It is known from that work also that TCNQ in  $\text{CH}_3\text{CN}$  reacts at 25°C with metallic Cu or Ag to form metal salts of the  $\text{TCNQ}^{\cdot-}$  anion radical on the metal. Films made in this way (1) are polycrystalline. In this work they are designated CP (for chemically prepared) films. The CP films studied had thicknesses in the 3-6  $\mu\text{m}$  range.

Homogeneous thin films, designated TF, of CuTCNQ and AgTCNQ were prepared by vapor deposition techniques as follows. On a clean substrate of Cu, Si, Al, KBr, or fused  $\text{SiO}_2$ , first a Cu (or Ag) film was vapor deposited under high vacuum, then a layer of TCNQ was deposited, and this was followed by deposition of alternating layers of Cu (or Ag) and TCNQ without breaking the vacuum at any point. The layer thicknesses were adjusted to produce a 1:1::Cu (or Ag):TCNQ mole ratio with both the bottom and top layers composed of the metal. In typical experiments the bottom and top Cu (or Ag) layers were 5nm thick and the final films were 0.2 to 0.8  $\mu\text{m}$  thick. After its formation, the multilayer structure was heated in situ (i.e.

without breaking the vacuum) or in an Ar-filled dry box at 100°C for 2 min. The resulting blue films were found to be only CuTCNQ (or AgTCNQ) by Raman spectroscopy. This was confirmed in each case, and in the case of films on SiO<sub>2</sub> (and other substrates that are transparent to visible light) by taking the Raman spectrum at both the top and, through the substrate, the bottom of the film (5).

The Raman spectra were measured on a Jarrell Ash 25-300 spectrometer with 90° scattering geometry using the 457.9, 476.5, 488.0, and 514.5 nm lines of a Spectra Physics 165 Ar-ion laser and the 632.8 nm line of a Spectra Physics 125 He-Ne laser. The spectral accuracy and resolution was 2 cm<sup>-1</sup> or better.

The partial and complete transformations of films were carried out by mounting the film (on its substrate) on an X-Y microscope translation stage and moving the film with this mechanism as it was exposed for controlled times to a fixed direction laser beam of controlled diameter, power, wavelength, and focus. The connections of the film elements into the circuits were made with Ag print paste. The electrical measurements were made using a Kiethley electrometer.

### 3. RESULTS AND DISCUSSION

The Raman spectra of the metal TCNQ materials described above were obtained with low source power before and after exposure to light powerful enough to cause changes in them.

#### 3.1 Partial Transformations

The Raman spectra of two types of CuTCNQ films, as prepared, are shown in Figure 1 (a) and (c). They were measured with a 488.0 nm source at low power (10 mW/mm<sup>2</sup> for the CP film and 20 mW/mm<sup>2</sup> for the TF film), and show the films to be pure CuTCNQ (1). The differences between the CP (a) and TF (c) spectra are due to resonance Raman phenomena. There is a small but significant

difference in their visible spectra which makes the TF spectrum essentially a normal (non-enhanced) Raman spectrum in which the relative intensities of non-totally symmetric modes are greater than in the preresonantly enhanced CP spectrum (6).

After irradiation of the CP film at  $50 \text{ mW/mm}^2$  for 10 sec with 488.0 nm light, its spectrum measured at low power at the same source wavelength is that shown in Figure 1 (b). Irradiation of the TF CuTCNQ film at  $200 \text{ mW/mm}^2$  for 10 sec at 488.0 nm caused transformation of the TF film such that it yielded the spectrum, measured at low power, shown in Figure 1 (d). Spectra 1 (b) and 1 (d) were selected to show different degrees of transformation. Comparison of the spectra of the irradiated samples 1 (b) and 1 (d) with those of the initial films, 1 (a) and 1 (c), and with the spectrum of pure TCNQ 1 (e) show that the transformation has produced materials whose spectra contain the features of both CuTCNQ and TCNQ (5). This conclusion is also supported by detailed resonance Raman studies (6). Spectrum 1 (b) of the transformed CP film was selected to show, by comparison with Figure 3 (b) of reference (1), that the optically induced transformation yields the same products as does the electric field-induced switching and can proceed to a greater extent in the transformed region.

The transformation of AgTCNQ is shown in Figure 2. There the spectra of a TF sample are shown initially 2 (a) and after partial transformation by exposure to 514.5 nm radiation at  $200 \text{ mW mm}^{-2}$  for 10 sec 2 (b). Clearly spectrum 2 (b) contains the features of both AgTCNQ and TCNQ itself. As in the case of CuTCNQ, the high reflectivity of the TF samples of AgTCNQ required that a rather high incident laser power be used for transformation, while the CP films and powders have much lower power thresholds. Thus, the

threshold for transformation of AgTCNQ powder by irradiation at 488.0 is  $3 \text{ mW mm}^{-2}$  while that for the AgTCNQ TF sample is  $150 \text{ mW/mm}^2$  at that wavelength.

The transformations reported above were accomplished with light of wavelengths less than 520 nm. Since the optical spectra of these metal TCNQ materials consists of two absorption band systems, one of which occurs at wavelengths longer than 520 nm, it was important to determine if transformation can be effected also at long wavelengths. The spectrum of AgTCNQ powder, measured at 632.8 nm with  $1 \text{ mW mm}^{-2}$  power, is shown in Figure 3 (a), and the spectrum of the powder measured similarly after irradiation at 632.8 at its threshold power of  $2.5 \text{ mW mm}^{-2}$  is shown in 3 (b). Clearly the transformation to a material containing both AgTCNQ and TCNQ is effected.

The electric field induced transformations of CuTCNQ and AgTCNQ occur fast (10 n sec) (2) on going to the low impedance state. The transformed materials return to the metal TCNQ spontaneously but, at 25°C, slowly. This constitutes a memory effect which also is exhibited in the case of the light-induced transformation. This is shown in Figure 4, in which 4 (a) is the spectrum of a transformed CuTCNQ CP film, taken 15 min after it had been irradiated above the threshold with 476.5 nm light, and 4 (b) is the spectrum of the same film measured after 2 hr. Clearly the photoinduced transformation exhibits the memory effect and the spontaneous return to the initial state. This return can be made slower by causing the transformation to proceed further. And, it can be made faster by raising the temperature of the metal and TCNQ mixture in the transformed region, as was shown by the TF preparation procedure. The recombination is thermally activated but the photoinduced transformation is not (7).

Films of both CuTCNQ and AgTCNQ which had been transformed partially, as described above, were washed with solvent and the spectra of the exposed

regions were measured again. In each case only the spectrum of the metal TCNQ was observed. In other experiments the exposed regions of analogously exposed films were removed by gentle scraping, under microscopic observation, rather than washing. Again the spectrum of the material beneath the surface layer was found to contain no TCNQ, it was that of only the untransformed metal TCNQ. Thus, the transformation under the conditions described occurs only in a surface layer. The implications of this for photoswitching phenomena will be considered below.

In order to determine whether such a transformation occurs in a material which contains TCNQ moieties in both a form giving the  $\text{TCNQ}^-$  spectrum and that giving the TCNQ spectrum (8),  $\text{Cu}(\text{TCNQ})_2$  was investigated. This material is important to study also because of the chemical nature of the transformation according to Eqn (I). Since the transformation in  $\text{CuTCNQ}$  produces TCNQ and returns to the untransformed state by recombination of the metal with this TCNQ, loss of TCNQ by sublimation would prevent full reformation. However, the stoichiometry of  $\text{Cu}(\text{TCNQ})_2$  provides a buffer against this effect.

The spectra in Figure 5 show the transformation of  $\text{Cu}(\text{TCNQ})_2$  stepwise from the initial material exhibiting the spectral features of both  $\text{TCNQ}^-$  and TCNQ, Figure 5 (a), through partial transformation, Figure 5 (b). The bands due to TCNQ and  $\text{TCNQ}^-$  are marked. The spectrum of the transformed powder, taken just before the last of the  $\text{Cu}(\text{TCNQ})_2$  had been transformed but before the TCNQ sublimed off, Figure 5 (c), shows that it is largely TCNQ, as shown by comparison with the spectrum of TCNQ itself, shown in Figure 5 (d).

In order to determine whether the electron transfer and accompanying processes could yield an analogous product with a metal of very low ionization potential,  $\text{LiTCNQ}$  powder was studied. In this case also TCNQ is formed as a result of the photoinduced transformation.

### 3.2 Complete Transformation

Two observations made in the course of this study led to our investigating the complete transformation of metal TCNQ materials, which is achieved when a reaches

$$\text{zero in (11) (9): } (M^+TCNQ^-)_a \xrightleftharpoons{\quad} (M^0)_y + y(TCNQ^0) \quad (11)$$

The first observation, shown in Figure 2 (c), was that the spectrum of a region on a AgTCNQ thin film becomes that of TCNQ<sup>0</sup> alone as it is being transformed photochemically. The second was that if any of these materials is placed under a cover glass and then the transformation is carried out, the cover glass acquires a film of TCNQ on it. These show that the transformation produces TCNQ which readily sublimes off of the sample. On the basis of these observations we concluded that the transformation can be driven to completion by a combination of light and removal of TCNQ to leave exposed regions containing only metal. Since this may have usefulness in forming metallic domains for optical or electronic applications, several types of experiments have been performed.

First, CuTCNQ and AgTCNQ TF samples were mounted on a microscope positioner and exposed to a focused beam of 488.0 nm radiation. Beam spots of 100  $\mu\text{m}$  to 200  $\mu\text{m}$  diameter and 200-250 mW power were used. The samples were moved under the beam. An optical transmission microphotograph of a 0.3  $\mu\text{m}$  TF AgTCNQ sample on SiO<sub>2</sub> exposed to a 120  $\mu\text{m}$  diameter beam scanned over it at ca 400  $\mu\text{m s}^{-1}$  is shown in Figure 6. The smallest separation of exposed regions on this sample is about 10  $\mu\text{m}$ . The exposed regions contain Ag but no TCNQ. Second, CuTCNQ TF samples were exposed to the same light beam directed to a region between electrodes separated by 0.3 to 0.6  $\mu\text{m}$ , the film thickness, and the resistance dropped to essentially the short circuit value for the system as expected for a metallic conduction path when complete transformation occurs.

### 3.3 Photoswitching Considerations

Since the partially transformed region is spectroscopically identical to electrically switched (low impedance) material in CuTCNQ or AgTCNQ films (1,2), but only a surface layer is transformed under the conditions used here, it is appropriate to ask whether the material will function as a photoswitch. This involves two separate questions; one, whether the films become conductive when phototransformed and the

field used to measure the conductivity is much lower than the electric field switching threshold, and two, whether they become conductive when the field is near but just below this threshold when the sample is exposed to light.

In the latter case the sharpness of the electric field threshold indicates that switching could occur but not be due to phototransformation alone. To analyze this case in light of the results reported here, consider the 5- $\mu\text{m}$  thick Cu/CuTCNQ/Al film reported (2,10) to undergo electric field induced switching at a threshold of 5.5 V (field strength of  $1.10 \times 10^6 \text{ V m}^{-1}$  (11)) and ask what would be the effect of phototransforming a 0.3  $\mu\text{m}$  thick layer just under a semitransparent Al electrode when a bias voltage of 5.4 V is applied. Assuming that this 0.3  $\mu\text{m}$  layer becomes conductive when it is phototransformed, the remaining high impedance, untransformed material would be a 4.7  $\mu\text{m}$  film in a  $1.15 \times 10^6 \text{ V m}^{-1}$  field. This is above the threshold for electric field induced switching, so switching to low impedance material would be expected.

These considerations lead to other possibilities, such as electric field induced switching with supra-threshold fields, and phototriggering of the electric field switching at lower light exposures. These will be discussed later (6), as will experiments on thin films phototransformed in the absence of electric fields or the presence of ones that are well below the electric field switching threshold.

#### Acknowledgment

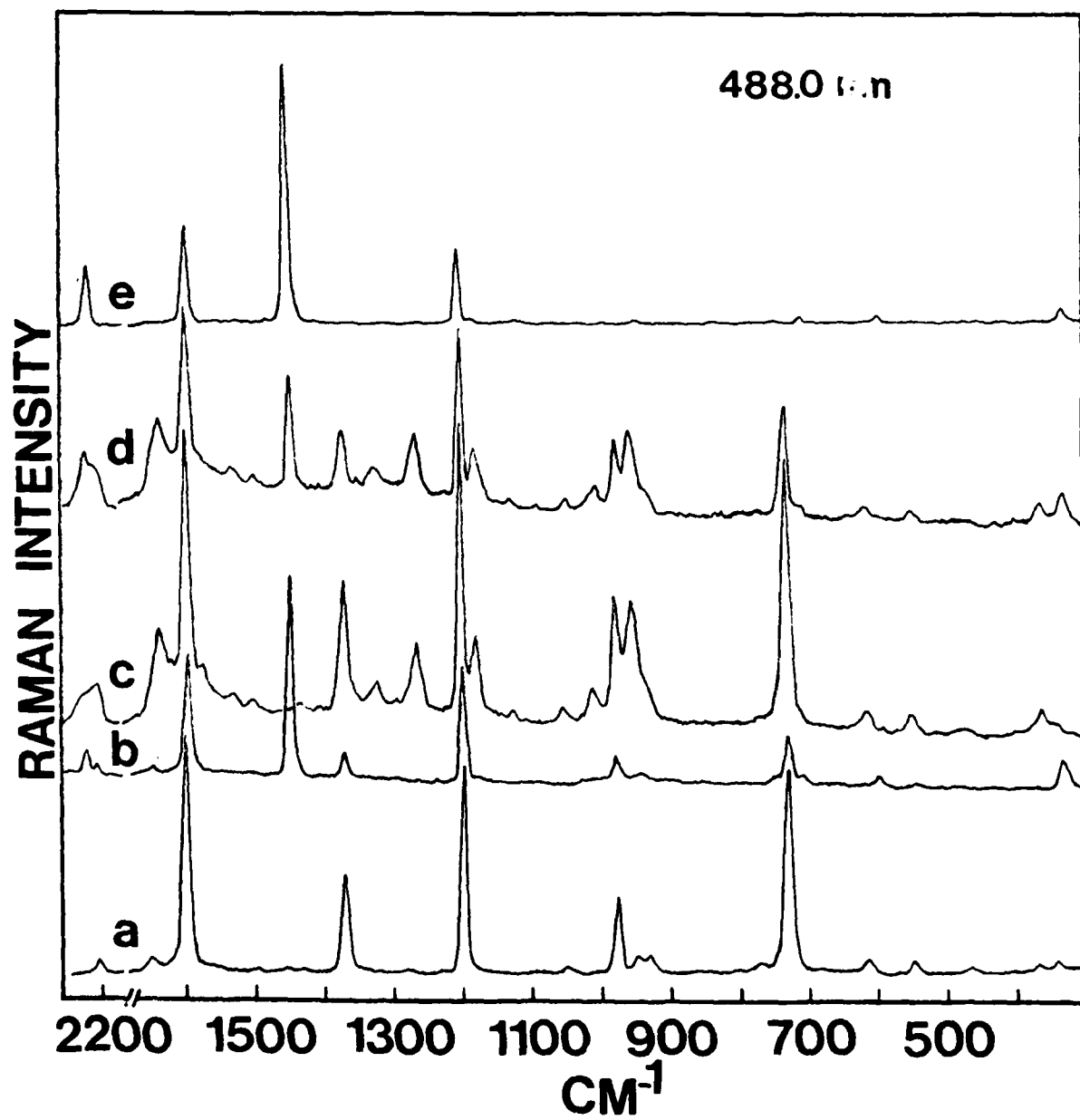
This work was supported by the Office of Naval Research and by the Materials Research Laboratory of Brown University through the National Science Foundation. We gratefully acknowledge discussions with Dr. Costas Tzimis of Bell Labs and Vincent Mattera of Brown University, and the kind assistance of Keith Warner, Herb Stanton and Ted Kirst of the MRL Facilities.

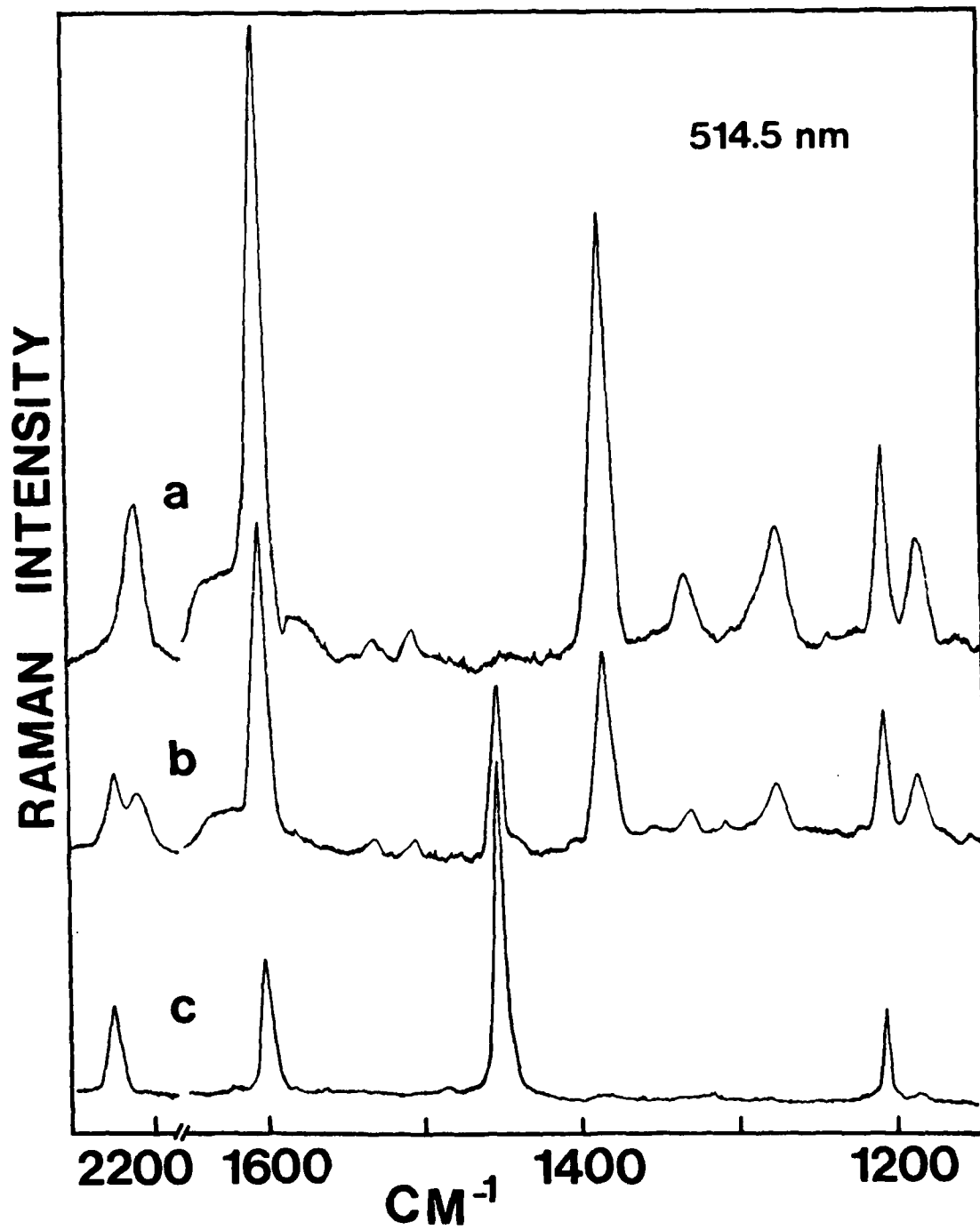
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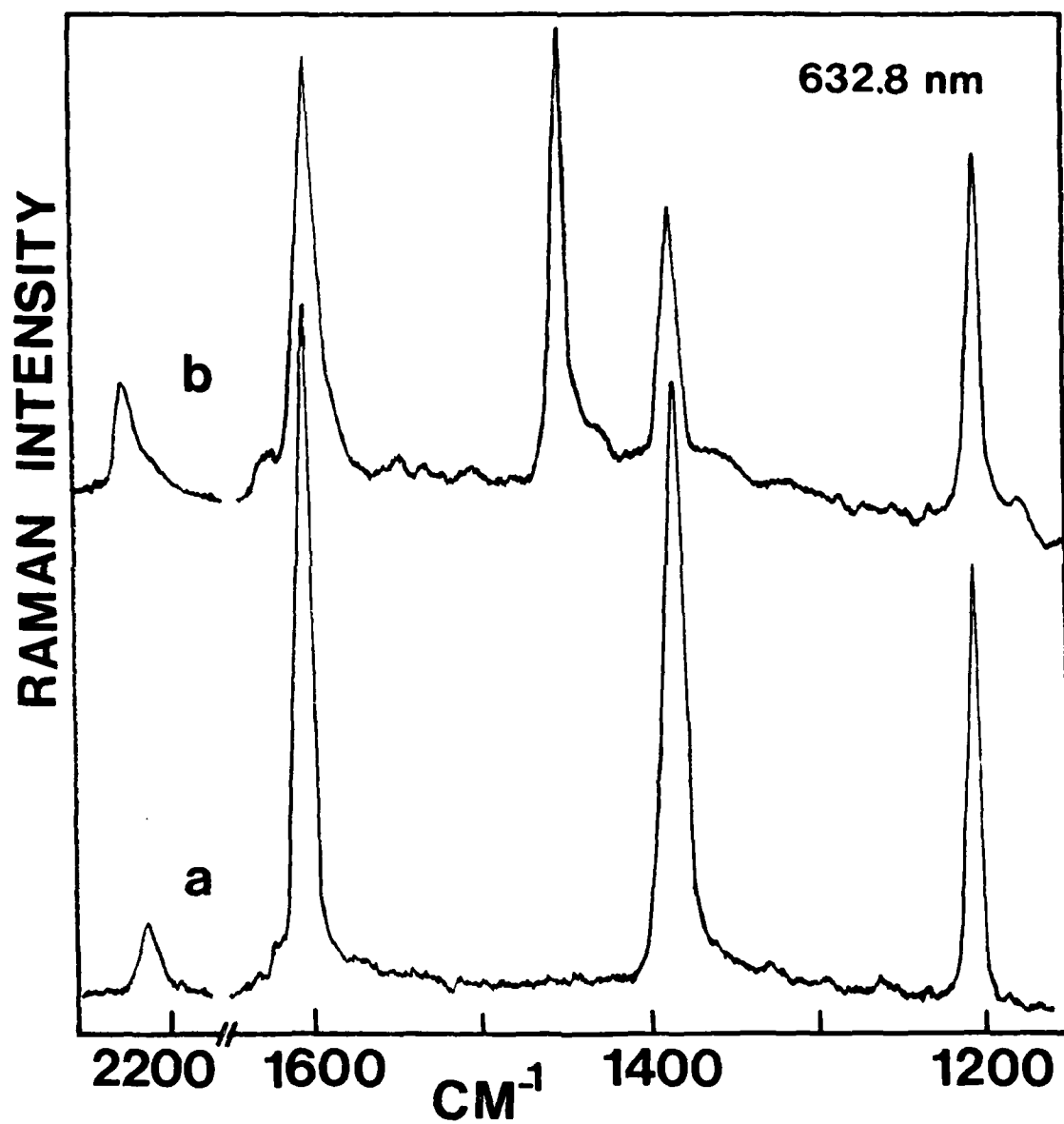
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4. L.R. Melby et al., J. Am. Chem. Soc. 84, 3374 (1962).
5. Key features of the Raman spectra which show the presence of TCNQ are the modes at 2223, 1451 and  $333\text{ cm}^{-1}$ , while the ones at 1598 and  $1204\text{ cm}^{-1}$  coincide with the corresponding modes of the  $\text{TCNQ}^-$  moieties.
6. E.I. Kamitsos, W.M. Risen, Jr., to be published.
7. (a) Raman spectra of  $\text{CuTCNQ}$  powder taken as a function of temperature gave no evidence of TCNQ production as a result of high temperature, even during the decomposition of the salt; (b) thermal decomposition studies (W.J. Siemons et al., J. Chem. Phys. 39, 3523 (1963) and Y. Kishimoto, W. Shimotsuma, Bull. Chem. Soc. Jpn, 53, 1723 (1980)) have shown that TCNQ salts of the type  $\text{M}^+(\text{TCNQ}^-)_x$ ,  $x = 1.5, 2.0$ , on heating dissociate into  $\text{M}^+\text{TCNQ}^-$  and  $\text{TCNQ}^0$ , while salts of the form  $\text{M}^+\text{TCNQ}^-$  decompose by losing the CN groups.
8. R. Bozio et al., J. Chem. Soc. Farad. Trans. II, 71, 1237 (1975).
9. Note that Eqn (I) is well defined only for partial transformations. Its left-hand side is the whole system before, and its right-hand side the whole system after partial transformation. To express a proper chemical equilibrium with the possibility of its being driven wholly to one side or the other, an equation such as (II) is required.
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11. As calculated from the film thickness and threshold voltage data given in (10); the reported value for the sample is  $4 \times 10^3\text{ V/cm}$ .

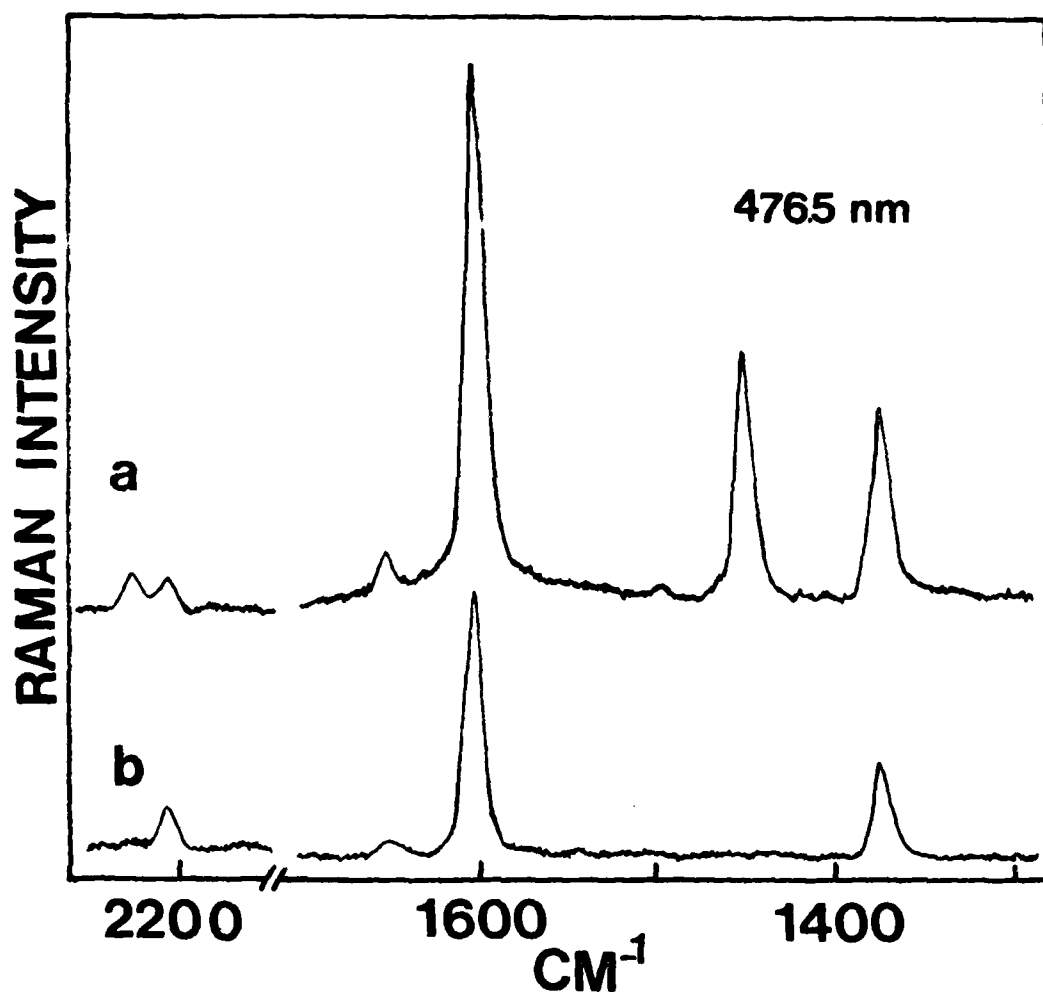
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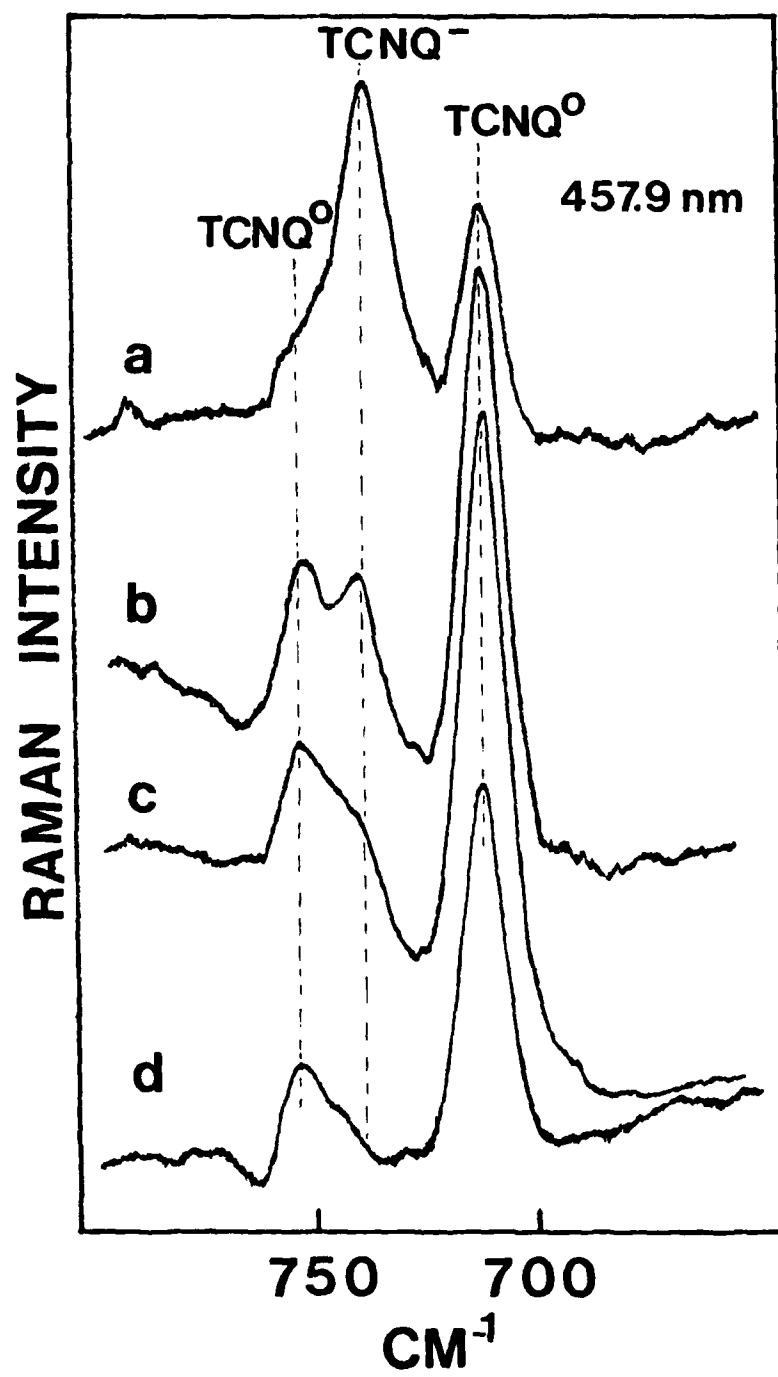
1. Raman spectra of (a) CuTCNQ CP film, untransformed (as prepared); (b) CuTCNQ CP film, partially transformed; (c) CuTCNQ TF film, untransformed; (d) CuTCNQ TF film, partially transformed, and (e) TCNQ. Designations and experimental conditions given in text.
2. Raman spectra of AgTCNQ TF films, (a) untransformed, (b) partially transformed, and (c) completely transformed.
3. Raman spectra of AgTCNQ powder (a) untransformed, and (b) partially transformed.
4. Memory effects in CuTCNQ CP film (a) partially transformed film, spectrum taken 15 min after transformation, (b) spectrum measured after 2 hr with low power.
5. Transformation steps in  $\text{Cu}(\text{TCNQ})_2$ , (a) initial material,  $\text{Cu}(\text{TCNQ})_2$ ; (b) partial transformation; (c) nearly complete transformation and (d) pure TCNQ.
6. Transmission optical microscope picture of a 0.3  $\mu\text{m}$  AgTCNQ TF on  $\text{SiO}_2$  showing optical writing performed with a 120  $\mu\text{m}$  diameter, 250 mW power, 488.0 nm laser source.

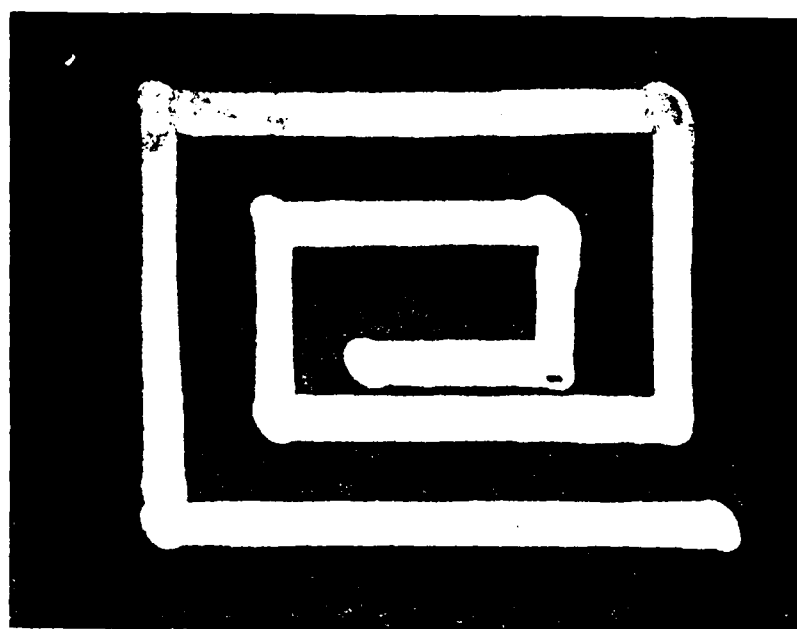












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